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INVESTIGATION OF COMPOSITE STRUCTURES FABRICATED WITH ADVANCED HIGH-STRENGTH, HIGH-MODULUS REINFORCEMENT MATERIALS

By

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August 1968

U. S. ARMY AVIATION MATERIEL LABORATORIES
FORT EUSTIS, VIRGINIA

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NORTON RESEARCH CORPORATION
CAMBRIDGE, MASSACHUSETTS

U. S. ARMY MATERIEL COMMAND

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The work reported herein was performed under Contract DAAJ02-67-C-0091 with the Norton Research Corporation (formerly National Research Corporation).

The data contained in this report are the result of research conducted to evaluate two concepts for producing boron thin-film reinforcement and composites.

The report has been reviewed by the U.S. Army Aviation Materiel Laboratories and is considered to be technically sound. It is published for the exchange of information and the stimulation of future research.

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Final Report

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SUMMARY

Previous work at Norton Research Corporation (NRC) demonstrated the feasibility of depositing boron by vacuum evaporation onto thin substrates such as aluminum foil and polyimide film. Sheets of these materials could then be bonded together with organic adhesives to form a multilayered composite. Laminates of this type had the advantage that the mechanical properties showed a high degree of isotropy in the plane of the laminate.

Early laminates had a relatively low volume fraction of reinforcement--approximately 20% boron. The present work is an investigation of two methods of improving the mechanical properties of the composites by increasing the volume fraction of reinforcement.

The technique used in the first method involved the deposition of boron on aluminum. Several sheets of this material were then bonded together with epoxy resin. Sodium hydroxide was used to dissolve the outer sheets of aluminum, thereby making primary composites with increased volume fractions of reinforcement. Primary composites were then relaminated to form a multilayered composite. A number of variations of this procedure were investigated. Volume fractions of up to 54% boron were achieved. This resulted in composites with moduli up to 31.1 x 10^6 psi and strengths up to 25.6 x 10^3 psi.

The second procedure involved the use of 1/4 mil polyimide film in place of the 1/2 mil material used previously. Five laminates containing volume fractions of up to 38.2% boron were made. Moduli up to 20.2 x 10^6 psi and strengths up to 39.6 x 10^3 psi were achieved. The low density of the composites results in high specific properties. The optimum values were: specific modulus, 3.3 x 10^8 inches; specific strength, 6.5 x 10^5 inches.

TABLE OF CONTENTS

	Page
SUMMARY	iii
LIST OF ILLUSTRATIONS	vi
LIST OF TABLES	viii
LIST OF SYMBOLS	ix
INTRODUCTION	1
SECTION I: TASK I PHASE	2
Evaluation of Concepts for Producing Composites From Thin Boron Films	2
Discussion of Results	7
Conclusions and Recommendations	11
SECTION II: TASK II PHASE	14
Material Preparation	14
Mechanical Testing of Composites	15
Discussion of Results	22
Conclusions and Recommendations	32
REFERENCES CITED	33
DISTRIBUTION	34

LIST OF ILLUSTRATIONS

Figure		Page
1	0-2 Arrangement	4
2	1-3 Arrangement	4
3	2-3 Arrangement	5
4	2-4 Arrangement	5
5A	1-2 Arrangement, Primary	6
5B	1-2 Arrangement, Secondary	6
6	Laminate Cutting Plan	17
7	Experimental Setup for Tensile Tests	18
8	Exploded Close-Up of Ball Bearing Swivel Shackle	19
9	Close-Up of Aluminum Gripping Cheeks for Tensile Specimens	20
10	Schematic Stress-Strain Curve for Composite Tensile Specimens, With Definitions of Elastic and Post-Elastic Parameters	21
11	Tensile Stress-Strain Curves for Boron/Polyimide Laminar Composite Specimens 40-114, A (0°), B (90°), and C (90°)	24
12	Tensile Stress-Strain Curves for Boron/Polyimide Laminar Composite Specimens 40-118, A (0°), B (90°), and C (90°)	25
13	Tensile Stress-Strain Curves for Boron/Polyimide Laminar Composite Specimens 40-120, A (0°) and B (90°)	26
14	Tensile Stress-Strain Curves for Boron/Polyimide Laminar Composite Specimens 40-122, A (0°) and B (90°)	27
15	Tensile Stress-Strain Curves for Boron/Polyimide Laminar Composite Specimens 40-124, A (0°) and B (90°)	28

Figure		Page
16	Polished Section at Fracture -- Specimen 40-118A	30
17	Polished Section at Fracture -- Specimen 40-120A	30
18	Effect of Adhesive Thickness on Tensile Strength	31

LIST OF TABLES

Table		Page
I	Summary of Laminate Details	8
II	Composite Properties	10
III	Calculated Values of Strain in Reinforcement . .	12
IV	Mechanical Properties of Boron/Polyimide (0.25 mil) Composites	23

LIST OF SYMBOLS

E_T = tensile modulus of elasticity (psi)

σ_{pl} = apparent proportional limit stress (psi)

σ_U = ultimate strength in tension (psi)

ϵ_{pl} = proportional limit strain

ϵ_f = strain at failure

V% = percentage of boron by volume

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INTRODUCTION

This report covers work performed by Norton Research Corporation under Contract DAAJ02-67-C-0091 with the U.S. Army Aviation Materiel Laboratories, Fort Eustis, Virginia. The research program was directed toward the evaluation of two concepts for the production of boron thin-film reinforcement composites.

The specific items of work for the contract were:

TASK I

1. Evaluate the following two concepts for producing boron thin-film reinforcement composites:

   a. Aluminum substrate removal.

   b. New thin plastic substrates.

2. Select and optimize the process parameters for the better of the two methods described in (1) above.

TASK II

1. Fabricate laminar composite test specimens by the optimum method.

2. Test a minimum of five tensile specimens in the 0° and 90° directions taken from these laminates, and test a minimum of five specimens for elastic modulus in the 0° and 90° directions.

3. Evaluate the data obtained from the test program.

The work covering the Task I section of the program is summarized first in Section I. That work indicated that the better of the two methods of producing boron thin-film composites was that relating to the use of thin plastic substrates--specifically the 1/4 mil polyimide film. Consequently, the fabrication and testing programs planned for Task II (described in Section II) used boron on 1/4 mil polyimide as the basic thin-film material for composite lay-up.

SECTION I: TASK I PHASE

EVALUATION OF CONCEPTS FOR PRODUCING COMPOSITES FROM THIN BORON FILMS

Aluminum Substrate Removal

Work prior to the present contract had shown that boron could be deposited by vacuum evaporation on aluminum foil or sheet. However, direct lamination of the boron-coated aluminum resulted in composites with relatively low volume fractions of reinforcement. One method of increasing the volume fraction which was shown to be feasible was to make primary laminates from 2 sheets of boron-coated aluminum by bonding the boron layers together with an epoxy adhesive. The aluminum could then be removed from the primary laminates by solution in caustic soda. However, complete removal of the aluminum left a very thin primary laminate which was difficult to handle and easily damaged. The main work of this part of the present program was then concerned with the testing and evaluation of procedures which would permit the fabrication of composites with high volume fractions of reinforcement and, at the same time, with developing practical procedures which would produce composites with increased specific strength and modulus.

Five related, but distinctly different, procedures of aluminum substrate removal were examined. Each of the procedures has been designated as a certain arrangement, such as a 1-3 arrangement. The numbers refer to the number of layers of aluminum and boron, respectively, in the primary or repeating element in a composite. For instance, a composite made by the 1-3 arrangement would contain, say, 18 layers of reinforcement made by relaminating 6 primary laminates, each of which contained 1 layer of aluminum and 3 layers of boron. The main features of each of the procedures examined are summarized below:

0-2 Arrangement

In this procedure, several attempts were made to successively build up a primary laminate by repeatedly bonding onto a sheet of boron-aluminum and removing all of the aluminum (see Figure 1). Attempts were made to provide increased support for the primary laminate during build-up by bonding the first layer to a thick metal plate with a temporary adhesive. This procedure was not successful. The temporary adhesive chosen was a high melting wax, and it did not give a stable bond during the epoxy curing cycle. It was also very time-consuming and difficult.

1-3 Arrangement

Two secondary laminates were made using this procedure (see Figure 2). The procedure had the advantage that the primary lay-up was relatively simple. In addition, it was not necessary to use thin aluminum for the outer layers. For instance, boron on 0.7 mil aluminum could be used for the central section of the primary laminate and boron on 2.0 mil aluminum could be used for the outer layers. Since the 2.0 mil aluminum was removed completely, only the 0.7 mil aluminum contributed to the final laminate composition. Since deposition on 2.0 mil aluminum is a simpler process than on 0.7 mil, the overall procedure is less restrictive.

2-3 Arrangement

This procedure (see Figure 3) is similar in principle to the 1-3 arrangement but includes a higher proportion of aluminum. This, in general, means that the laminate is easier to make and handle, but this gain is at the expense of reduced volume fractions of reinforcement. One secondary laminate was made using this procedure.

2-4 Arrangement

This procedure is similar to the 2-3 arrangement (see Figure 4). The possible volume fractions of reinforcement are higher than the 2-3 but not as high as the 1-3 arrangement. Two secondary laminates were made by this procedure.

1-2 Arrangement

Two variations of this procedure were examined. In the first of these, an uncoated sheet of 0.7 mil aluminum was used as a central aluminum layer (see Figure 5). In the second case, the central aluminum sheet in the primary laminate was 0.3 mil thick. The main purpose of the central aluminum layers was to give some strength to the primary units so that they could be handled without damage for the lay-up of the secondary laminate. Uncoated aluminum was used because it was flatter and less wrinkled than boron-coated aluminum. The procedure also had the advantage that the boron could be deposited on a relatively thick aluminum substrate, e.g., 2.0 mil, without decreasing the volume fraction of reinforcement in the final laminates because all of the substrate aluminum was removed in making the primary unit. Two laminates were made by this procedure, one with a 0.7 mil core and one with a 0.3 mil aluminum core.

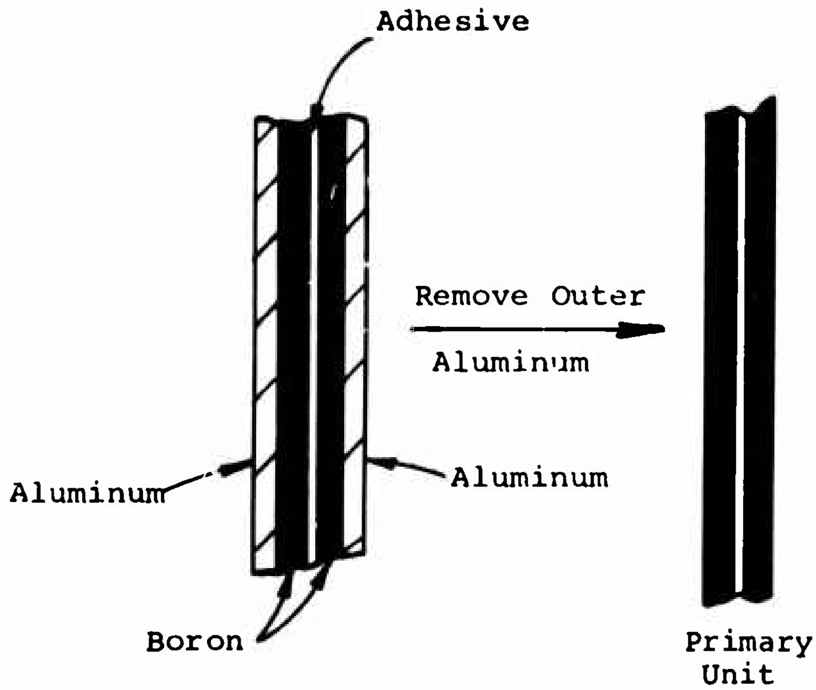

Figure 1. 0-2 Arrangement.

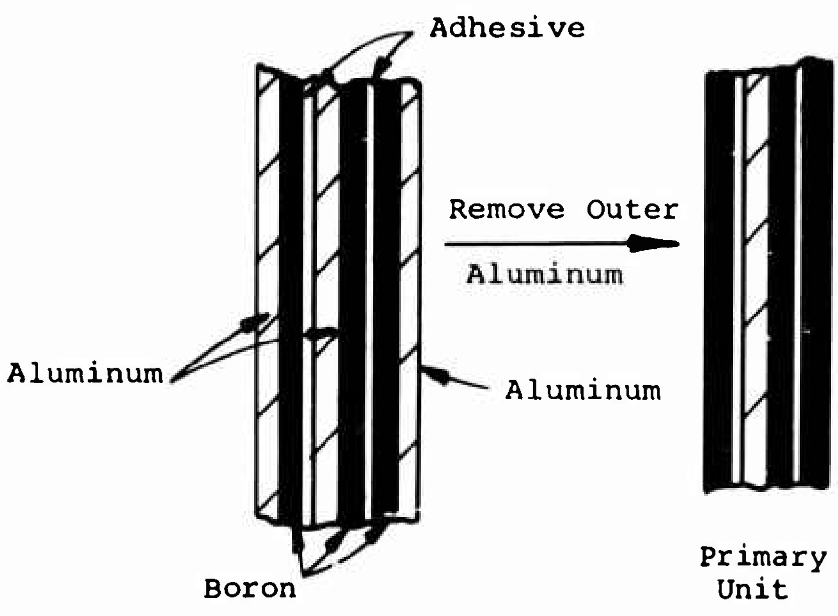

Figure 2. 1-3 Arrangement.

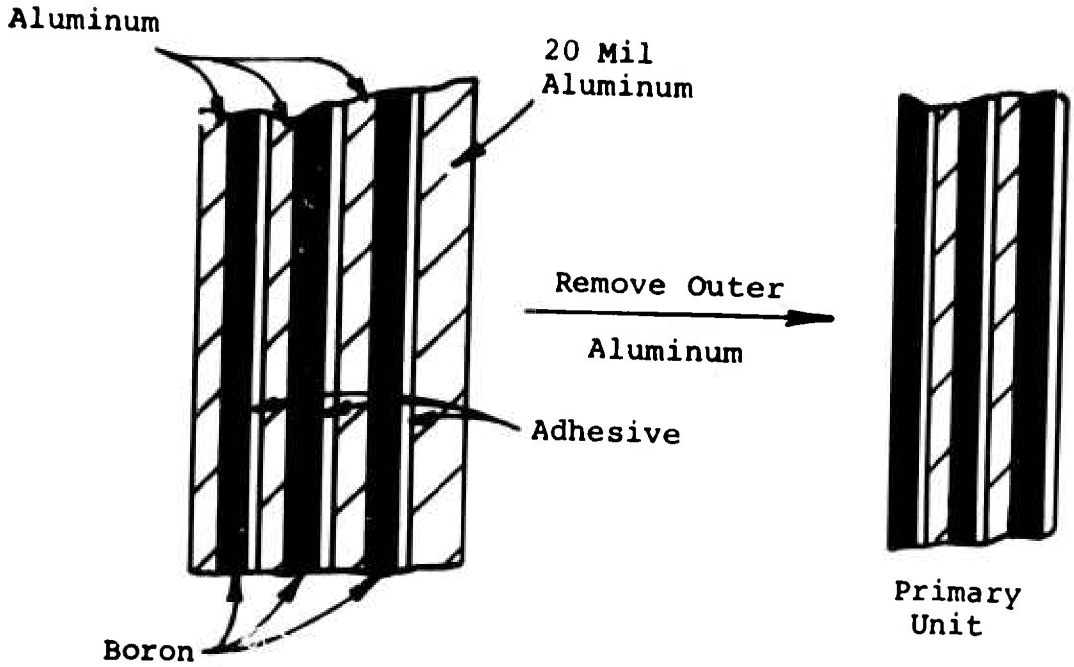

Figure 3. 2-3 Arrangement.

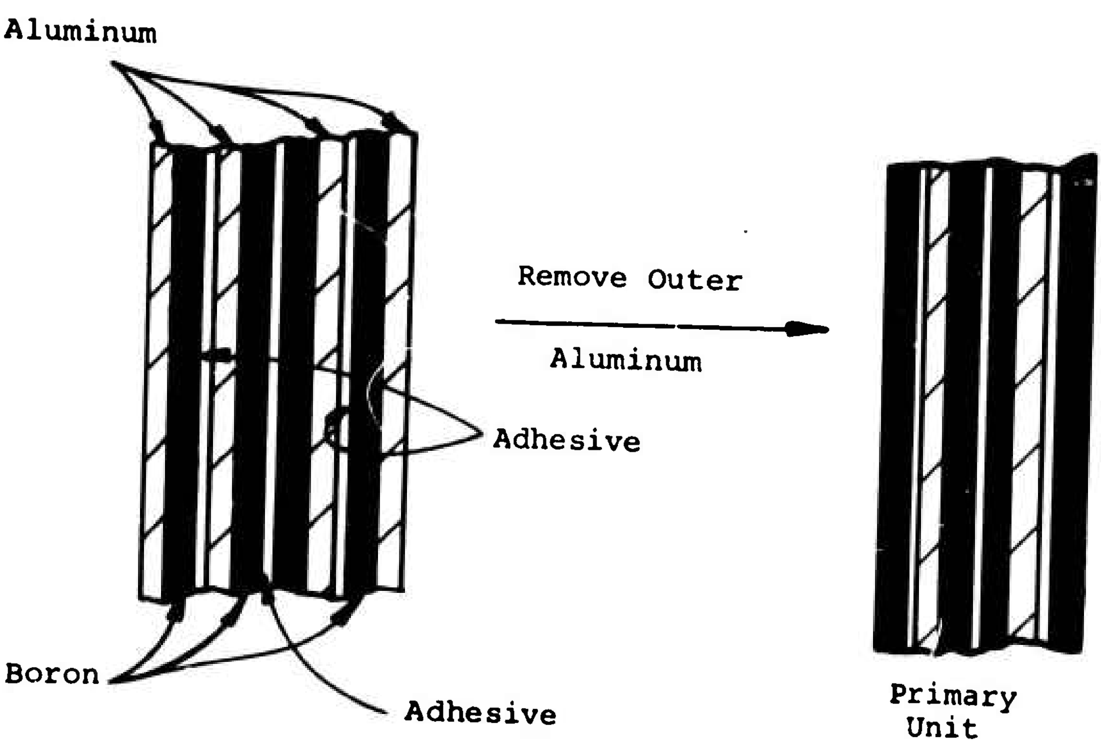

Figure 4. 2-4 Arrangement.

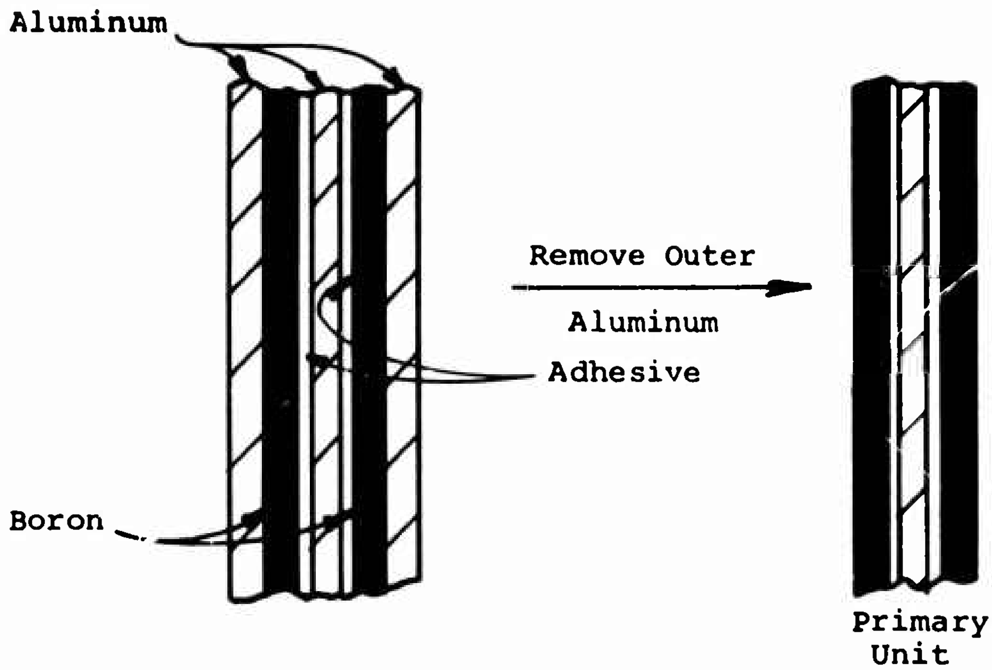

Figure 5A. 1-2 Arrangement, Primary.

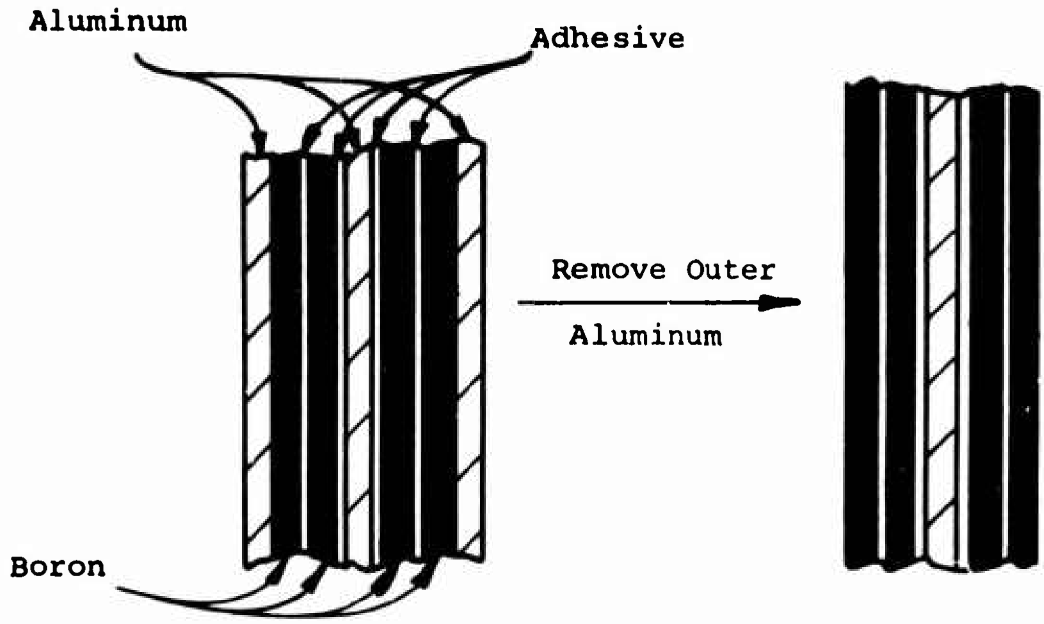

Figure 5B. 1-2 Arrangement, Secondary.

In general, all the substrate removal techniques called for a considerable amount of effort and care during lay-up, and they all required at least two main laminating steps. The primary laminates were, in general, very fragile, particularly those with the higher volume of reinforcement. Where the volume fraction of aluminum was relatively high in the primary laminate, say, the 2-3 arrangement, the primary laminate was less fragile, but then the volume fraction of reinforcement was considerably reduced.

Composites From Thin Plastic Substrates

The second procedure for increasing the volume fraction of reinforcement was based on the use of substrate materials which were thinner than the 0.5 mil polyimide used previous to this contract. Two materials were investigated. The first was 1/4 mil polyimide which was available in experimental quantities. The second was polyimide resin which was deposited from solution onto 2 mil aluminum. Experimental work on the 1/4 mil polyimide was extended to the production of one primary laminate and two secondary laminates. Work on the formation of polyimide films bonded to aluminum was carried to the stage that it was shown that films could be made and coated with reinforcement. However, no laminates were made by this procedure. Work on the 1/4 mil had shown that this was a promising approach, and efforts were directed to use it for the basis of the comparison with the substrate removal techniques.

The details of the composites made by the substrate removal techniques and with the use of the 1/4 mil polyimide are shown in Table I. The details of the mechanical test results of the laminates made are shown in Table II.

DISCUSSION OF RESULTS

The data in Table II indicate that composites with volume fractions of reinforcement of between 27% and 54% were made during this program. The low volume fractions for composites 40-63 and 40-80 reflect the larger amounts of aluminum associated with the 2-3 and 2-4 arrangements.

The values of the tensile modulus for the composites range from 16.5×10^6 to 31.1×10^6 psi. In general, these values are roughly proportional to the volume fraction of boron. A modulus of 31×10^6 psi and a density of 0.081 pci give a specific modulus of 3.8×10^8 inches. This value is 3.8 times that of aluminum.

By using Law-of-Mixtures calculations and a knowledge of the composition of the laminates, it is possible to calculate the

TABLE I. SUMMARY OF LAMINATE DETAILS

A. Composites by Substrate Removal Techniques	Composite No.
1. 0-2 Arrangement	
a) Heavy Metal Plate Base Premature Release From Base Stopped at 0-2 Stage	
b) Thin Aluminum Sheet Base (20 mil) Caustic Attack of Base Stopped at 0-2 Stage	
2. 1-3 Arrangement	
Two Secondary Laminates	
20 Layer (Six 3-layer primary laminates + 2 cover layers)	2844-73
14 Layer (Four 3-layer primary laminates + 2 cover layers)	40-67
3. 2-3 Arrangement	
One Secondary Laminate	
14 Layer (Four 3-layer primary laminates + 2 cover layers)	40-63
4. 2-4 Arrangement	
Two Secondary Laminates	
18 Layer (Four 4-layer primary laminates + 2 cover layers)	40-80A 40-80E
5. 1-2 Arrangement (0.7 mil Al)	
One Secondary Laminate	
14 Layer (Four 3-layer primary laminates + 2 cover layers)	40-102
6. 1-2 Arrangement (0.3 mil Al)	
One Secondary Laminate	
11 Layer (Three 3-layer primary laminates + 2 cover layers)	40-104

TABLE I - Continued

B. Composites From Thin Plastic Substrates	Composite No.
Polyimide Film - 1/4 mil Thickness	
One Primary Laminate	
20 Layer	40-76
Two Secondary Laminates	
160 Layer for Shear Test	40-76E
160 Layer for Compression Test	40-76C

TABLE II. COMPOSITE PROPERTIES

Type of Primary Laminate	Composite Material (4)	Composite Number	Density (pci)	Boron V%	Type of Test	Tensile Modulus 10^6psi	Theoretical Tensile Modulus 10^6psi (1)	σ_{pl} 10^3psi (2)	σ_{max} 10^3psi (3)
A. Substrate Removal									
1-3	B/$Al_{0.7}$	2844-73A	.0774	44	Uni. Ten.	22.2	27.7	10.1	15.8
1-3	B/$Al_{0.7}$	2844-73B(5)	.0774	44	Uni. Ten.	24.2	27.7	11.2	18.2
1-3	B/$Al_{0.7}$	40-67	.0814	54	Uni. Ten.	31.1	33.1	8.2	23.2
2-3	B/$Al_{0.7}$	40-63	.0852	47	Uni. Ten.	28	29.6	14.0	25.6
2-4	B/$Al_{0.7}$ B/$Al_{2.0}$	40-80	.080	27	Uni. Ten.	16.5	19.9	6.3	14.7
1-2	B/$Al_{2.0}$ +$Al_{0.7}$	40-102	.0784	30	Uni. Ten.	20.3	20.9	10.5	25.6
1-2	B/$Al_{2.0}$ +$Al_{0.3}$	40-104	.0794	50	Uni. Ten.	28.8	30.6	13.0	23.6
B. Thin Substrate									
1-1	B/$E_{0.25}$	40-76	.0574	34	Uni. Ten.	17.4	18.7	15.9	29.5
		40-76		34	Compression				47.7
		40-76		34	Interlam. Shear				>4.83

(1) Calculated from Law-of-Mixtures using boron modulus 55 x 10^6 psi and aluminum modulus 10 x 10^6 psi.
(2) Stress at proportional limit.
(3) Maximum tensile stress before failure.
(4) Subscript is thickness of aluminum in mils.
(5) Second test specimen from composite 2844-73.

effective modulus of the boron in the composites. The values range from 42 to 54 x 10^6 psi. The lower values represent a translation of about 75% of the theoretical modulus of boron into the composite, while the highest value is close to theoretical.

The maximum ultimate tensile strength obtained was 29.5 x 10^3 psi for the thin substrate laminate, 40-76. The lowest ultimate strength was 14.7 x 10^3 psi for the 2-4 arrangement. The proportional limit values also showed a considerable variation, from 6.3 x 10^3 psi to 15.9 x 10^3 psi. Separate work at NRC has indicated that the departure from linearity at the proportional limit stress is associated with the initial failure of the reinforcement. Consequently, in a number of instances the reinforcement began to fail at very low strains--less than 0.05% (see Table III). The highest value of the failure strain was obtained from the thin plastic substrate composite, 40-76. The thin plastic substrate composites also exhibited the least density.

Company-sponsored work at NRC, concurrent with the present program, has shown that, in general, boron deposited on aluminum has a low failure strain. It is probable that there is interaction between the aluminum and boron to form solid solutions and various aluminum boride compounds. If aluminum boride is formed, it is possible that this contributes to low failure strains since it is graphitic in form.

CONCLUSIONS AND RECOMMENDATIONS

The work in Task I showed that high volume fractions of reinforcement could be achieved by substrate removal techniques and that high specific moduli were attainable. On the other hand, the composite strengths obtained using the substrate removal techniques were low. In addition, the procedures were complicated, time-consuming, and probably not adaptable to automatic handling and scale-up.

The work on the thin substrates, on the other hand, showed definite promise. Although the volume fraction obtained in 40-76 was not as high as the highest substrate removal composite, the likelihood of improvement was high. The maximum strength obtained (29.5 x 10^3 psi) was the highest value obtained to that stage in the program, and it was also higher than any obtained on the thicker (0.5 mil) polyimide substrates in other work at NRC. The lay-up of composites based on the thin substrate was relatively easy, and an important feature was that this type of composite typically has a low density.

TABLE III. CALCULATED VALUES OF STRAIN IN REINFORCEMENT AT FAILURE

Primary Laminate	Material	Composite No.	Failure Strain %
A. Substrate Removal			
1-3	$B/Al_{0.7}$	2444-73	0.045
1-3	$B/Al_{0.7}$	40-67	0.026
2-3	$B/Al_{0.7}$	40-63	0.050
2-4	$B/Al_{0.7}$	40-80	0.038
1-2	$B/Al_{0.7}$	40-102	0.052
1-2	$B/Al_{0.3}$	40-104	0.045
B. Thin Substrate			
	$B/H_{0.25}$	40-76	0.091

The thin substrate method was therefore selected for the fabrication of specimens for Task II.

The plans were:

1. Coat 1/4 mil polyimide with boron.

2. Make 5 laminates.

3. Test these laminates in the 0° and 90° directions for both strength and elastic modulus.

4. Evaluate and report data.

The work completed in Task II is described in the following section of this report.

SECTION II: TASK II PHASE

MATERIAL PREPARATION

Boron was vacuum deposited on 1/4 mil polyimide film using the application and technique described previously.[1,2] In general, a thin coat (approximately .05 mil) was deposited on one side of the substrate and a thicker coat (approximately 0.2 mil) on the other.

The coated material was inspected, and high-quality sheets were selected for fabrication. Measurements were made to determine the average thickness of the deposit. Samples were also taken and boiled in water for 2 hours. Following this, the dry material was subjected to a "Scotch Tape" adhesion test. Only material which showed good adhesion between the deposit and substrate after a 2-hour water boil was used for making laminates.

The boron-coated material was then cut to size for laminate lay-up. In general, the laminate size was 5 inches x 4 inches, made from sheets 8 inches x 4 inches. This permitted 1.5 inches of each sheet to extend out beyond the ends of the pressed section. These parts of the film were used to hold the laminate sheets and to prevent them from moving too much during the laminating operations.

The typical laminate was 20 layers thick; consequently, 19 layers of adhesive were required for bonding. The resin system used was Union Carbide ERL-2256 together with curing agent Z. Vacuum laminating techniques were applied as outlined in the following procedure:

1. Material to be laminated is sampled, weighed, cut to size, and measured.

2. Sheets are then cleaned by thorough spray rinsing with warm acetone followed by hot alcohol.

3. When dry (a few minutes in air), the sheets are wet spray coated with an adhesive mixture of ERL-2256 (100 parts by weight) and curing agent Z (20 parts by weight) diluted with 9 times its volume of methyl ethyl ketone.

4. The sheets are then drained in air for 5 minutes and hung in an air oven at 225°F for 45 minutes.

5. The precoated sheets are then wet spray coated with the same adhesive mixture diluted with an equal volume

of methyl ethyl ketone.

6. The sheets are then drained in air for 5 minutes and hung in an air oven at 225°F until tacky--approximately 20 minutes.

7. The sheets are then laid up with a cross bead of undiluted adhesive mixture between each layer. Each bead contained 1-2 cc. of resin.

8. The laminate package is then evacuated to less than 200 microns for 20 minutes without applying any pressure to the laminate.

9. Pressure is slowly applied to the package and brought up to 500 psi.

10. Heat is applied to cure the adhesive. Cure conditions used are 1 hour at 180°F followed by a post-cure of 1 hour at 300°F.

All laminates with the exception of 40-124 were made up so that the shorter dimension (4 inches) corresponded to the transport direction of the 9-inch-wide continuous length of polyimide film through the coating operation. This has been defined as the 0° direction. The 90° direction then refers to specimens taken along the length of the laminate (5 inches) and therefore across the width of the main length of polyimide film as transported through the coating operation.

In the case of sample 40-124, the laminate size was increased to 5 inches x 5 inches and the laminate was laid up in cross ply; that is, 0°, 90°, 0°, 90°.

After completion of the post-cure, the press was water cooled and the laminate was removed, trimmed, and weighed. Accurate size and weight measurements were then made to permit the calculation of average density, glue line thickness, etc.

The laminates were then ready for the preparation of test specimens.

MECHANICAL TESTING OF COMPOSITES

In order to prepare test specimens with accurate dimensions, the following was used:

The laminate plate was fixed to a "Transite" (asbestos-cement composition) board with melted beeswax. The desired cuts were made on a "Delta" surface grinder with a 6-inch diamond slitting wheel, 220 grit, rotating at about 3,400

rpm. The longitudinal feed rate was about 1 inch per minute. Kerosene spray mist was used as a coolant at first, but a commercial spray coolant was later substituted to eliminate a possible fire hazard. The cut resulted in smooth edges with no edge cracking visible under optical examination. After cutting, the individual specimens were removed from the "Transite" base by gently warming the wax, and the wax remnants were washed from the specimens with trichloroethylene.

Most of the laminate plates, measuring originally 5 inches x 4 inches, were subdivided as shown in Figure 6.

The specimens were subjected to the following tests:

Tensile Strength

Tests to determine the ultimate strength in uniaxial tension of specimens 3/4 inch wide, about 0.010 inch thick, and about 4 inches long were made. The tensile specimens had a uniform cross section throughout, and they were gripped with aluminum cheeks fastened with adhesive at the ends. No lateral pressure was exerted on the specimens; the load transfer was entirely by shear traction. The gage length was 1 inch. Ball bearing swivels were incorporated into the cross-head and suspension of an Instron testing machine to relieve any initial misalignment and eccentric loading. The experimental setup is illustrated in Figure 7. An "exploded view" close-up of the ball bearing swivel shackle is given in Figure 8. Figure 9 shows the hyperbolically tapered profile of the aluminum gripping cheeks and a typical fracture in mid-gage length of a tensile specimen. All specimens fractured at or near the middle of the gage length.

Tensile Modulus

Tensile modulus was derived from the initial slope of the stress-strain curve generated in uniaxial tension tests. Strain was measured with SR-4 strain gages mounted directly on the specimen surfaces. Such strain gages were used in pairs in order to compensate for any bending components. Elastic displacements of the testing machine and in the tensile grips did not interfere with the strain measurements. Load strain curves for each test were generated by simultaneously plotting the output signals from the Instron load cell and from the strain gages on an X-Y recorder. Continuous curves from the initial linear rise, through the apparently "plastic" range, to the point of fracture, were obtained and converted to engineering stress-strain curves by dividing all values of load by the initial cross sectional area.

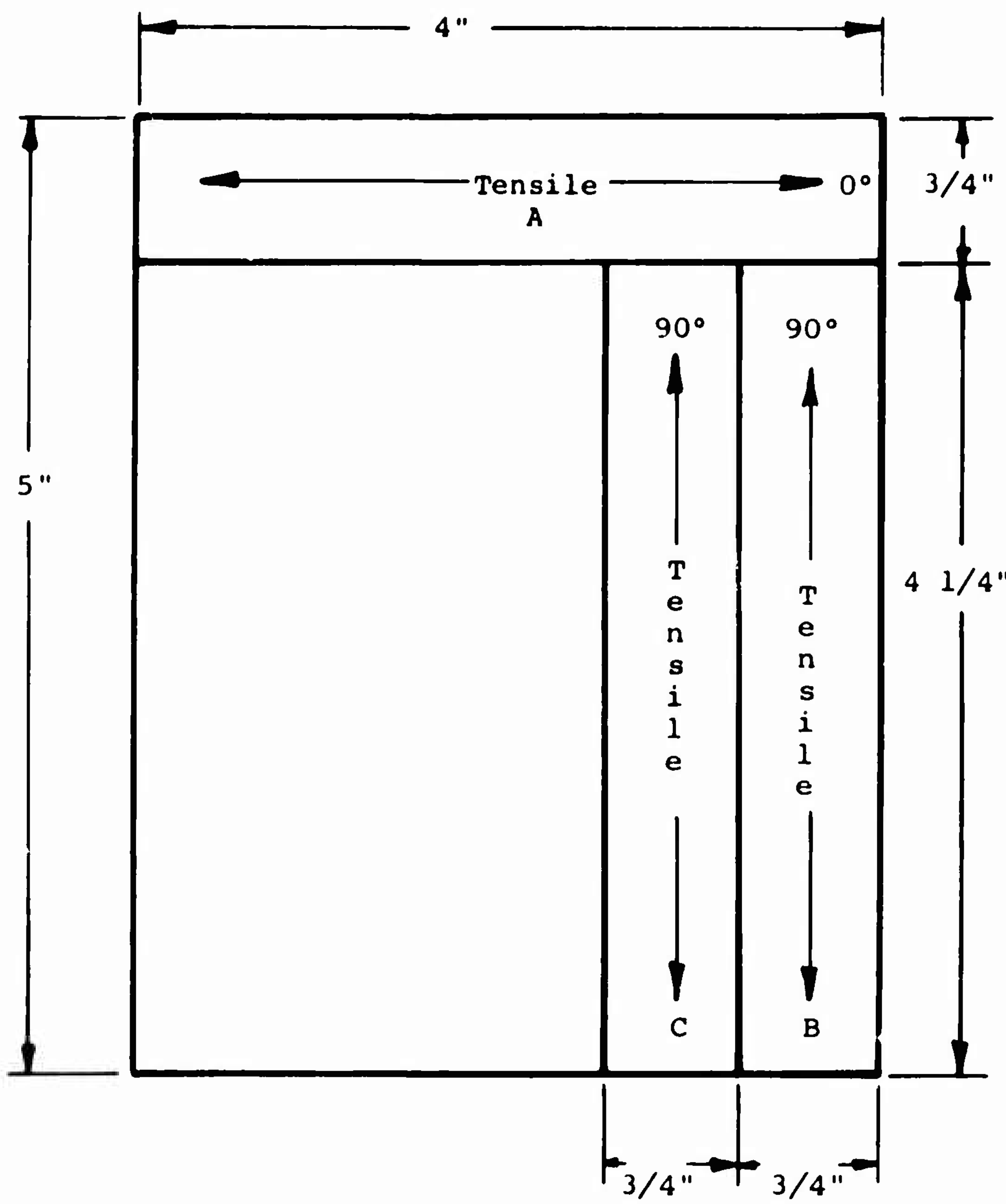

Figure 6. Laminate Cutting Plan.

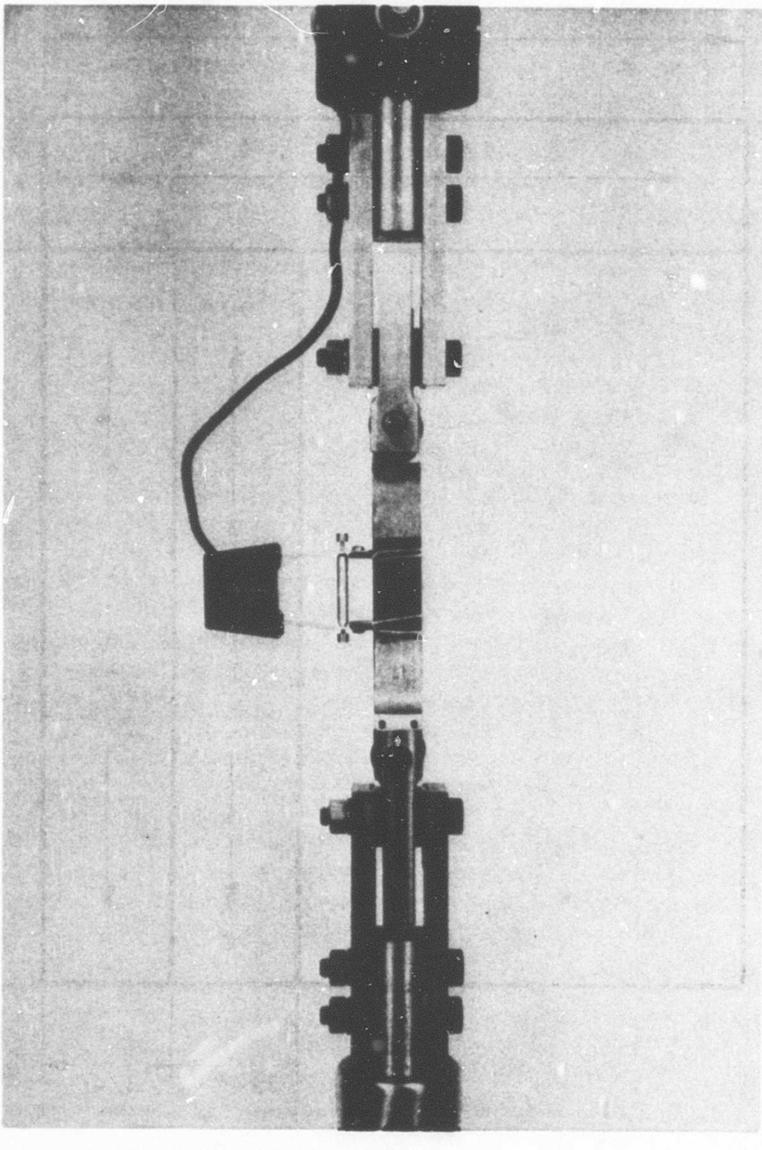

Figure 7. Experimental Setup for Tensile Tests. (Extensometer Not Used in Present Program).

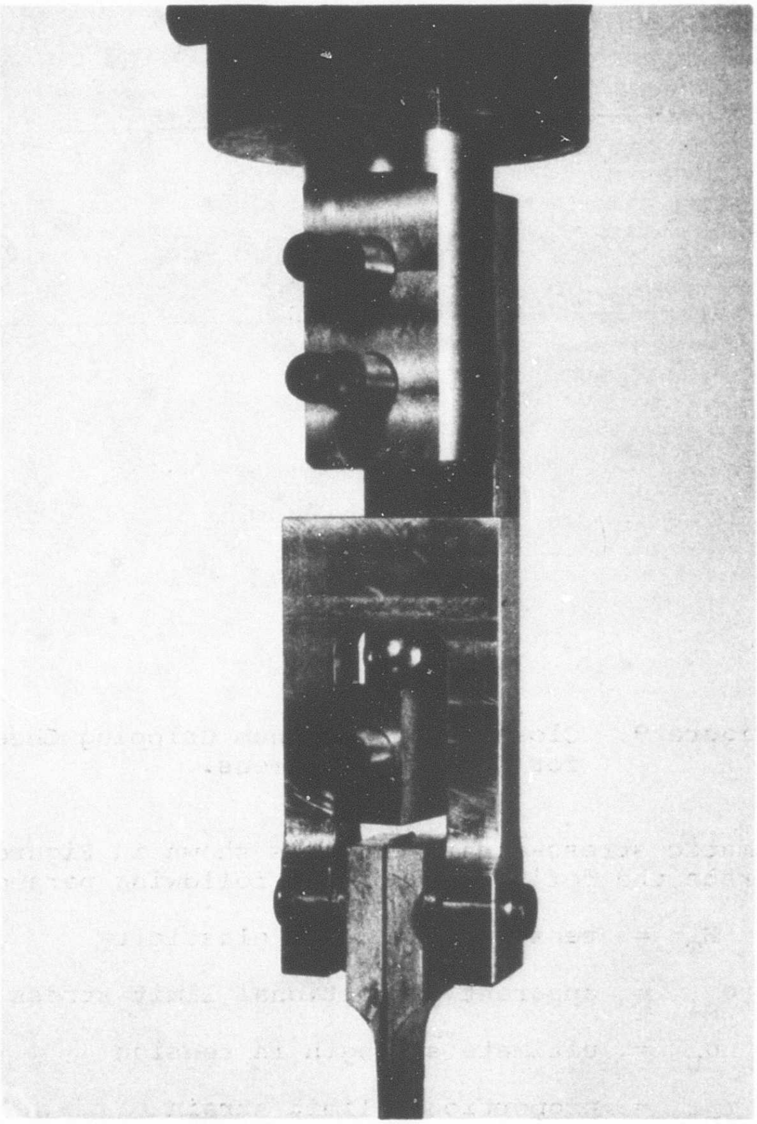

Figure 8. Exploded Close-Up of Ball Bearing Swivel Shackle.

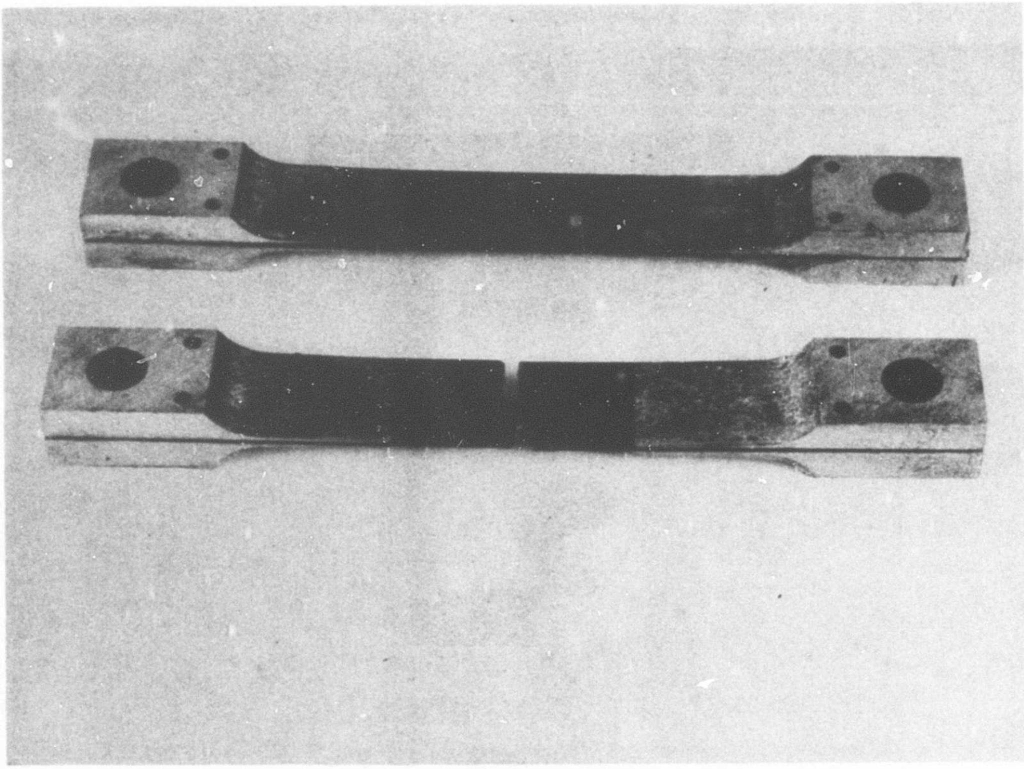

Figure 9. Close-Up of Aluminum Gripping Cheeks for Tensile Specimens.

A schematic stress-strain curve is shown in Figure 10 to illustrate the definitions of the following parameters:

E_T = tensile modulus of elasticity

σ_{pl} = apparent proportional limit stress

σ_U = ultimate strength in tension

ϵ_{pl} = proportional limit strain

ϵ_f = strain at failure

Following the tensile test, selected specimens were mounted and microscopically examined. Samples were also taken for chemical analysis of the boron content of the specimens.

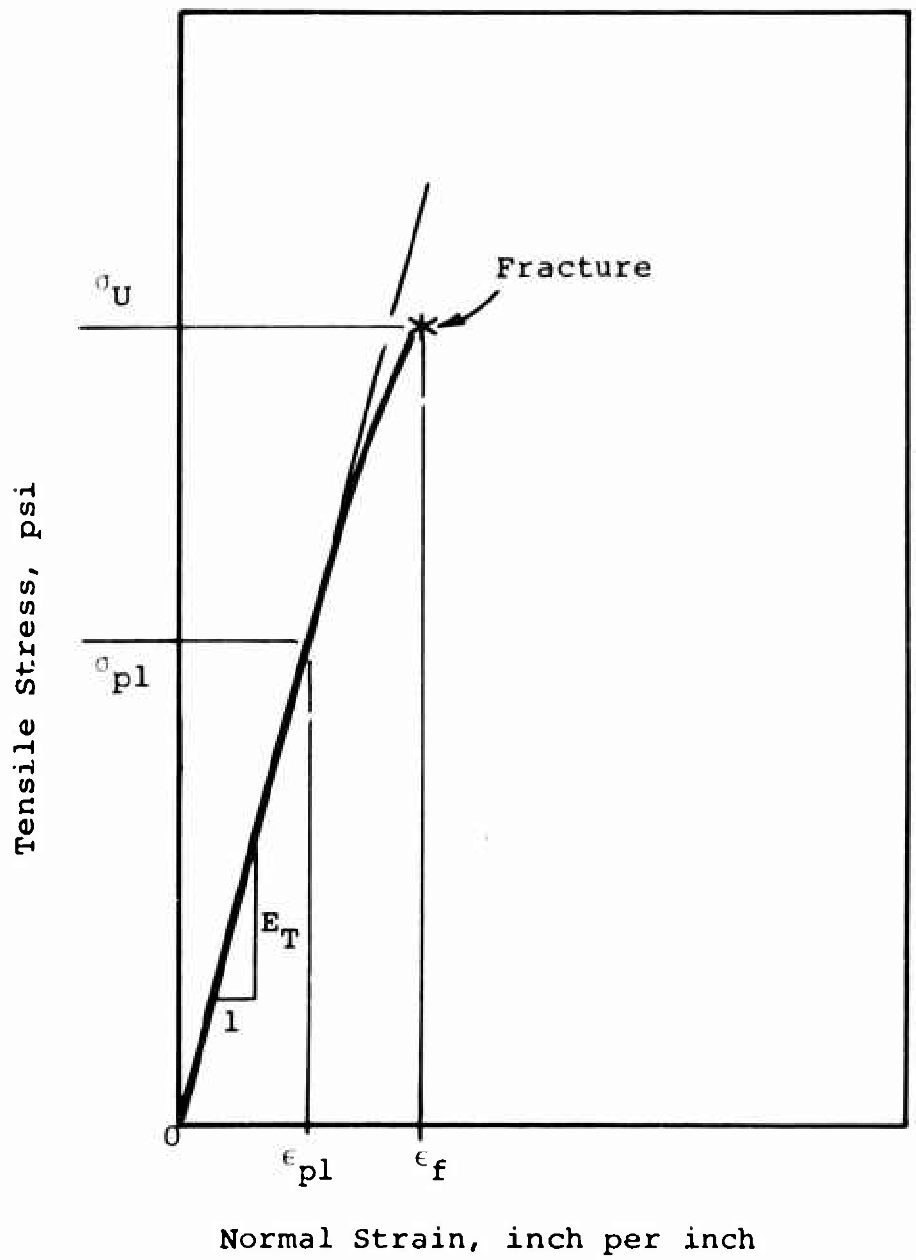

Figure 10. Schematic Stress-Strain Curve for Composite Tensile Specimens, With Definitions of Elastic and Post-Elastic Parameters.

Results

The major test results are summarized in Table IV.

The load-strain curves have been reproduced as stress-strain curves in Figures 11 through 15.

As shown in Table IV, a single determination of density was made for each laminate. In some cases the A sample was measured and in some cases the B sample. It has been assumed that the density differences between samples A, B, and C were relatively small. The density numbers in parentheses in Table IV are assumed values taken to be the same as the measured values of other specimens from the same laminate. Chemical analyses were made for each test specimen. These analyses quoted in parentheses in Table IV reflect the use of the assumed density values in calculating the volume fraction of reinforcement.

DISCUSSION OF RESULTS

A number of significant points are apparent on examining the data:

1. The tensile moduli varied from 9.3×10^6 psi for 40-118A to 20.2×10^6 psi for 40-120A. Similarly, the ultimate strengths ranged from 18.0×10^3 for 40-118B to 39.6×10^3 psi for 40-120A.

2. The upper values of these properties, namely, a modulus of 20.2×10^6 psi and a tensile strength of 39.6×10^3 psi, were the highest values which have been obtained with polyimide-based laminates to date. They represent marked increases in properties over those obtained in the Task I phase of the contract (modulus 17.4×10^6 psi, tensile strength 29.5×10^3 psi).

3. In general, the A specimens in each laminate showed higher ultimate strength values than either the B or the C specimens. However, in the case of laminate 40-126, the difference was small. In this laminate, the individual sheets of the laminate were alternated from 0° to 90°.

4. On the other hand, the degree of variation between specimens A, B, and C within any one laminate in the case of modulus and the proportional limit stress was relatively small. The initial slopes of the stress-strain curves and the initial departure from linearity

TABLE IV. MECHANICAL PROPERTIES OF BORON/POLYIMIDE (0.25 mil) COMPOSITES

Spec. No.	Density (pci)(1)	Boron Vol (%)(2)	Elastic Modulus 10^6psi	σ_{pl} Modulus 10^3psi	σ_{Max} Stress 10^3psi	Failure Strain (%)	Test Angle (deg)
40-114A	.061	37.4	20.1	20.1	35.4	.225	0
40-114B	(.061)	(38.1)	19.45	18.7	24.4	.145	90
40-114C	(.061)	(38.25)	19.05	13.96	25.3	.165	90
40-118A	.0498	18.8	9.3	11.3	20.1	.42	0
40-118B	(.0498)	(21.1)	12.6	11.62	18.0	.254	90
40-118C	(.0498)	(19.8)	9.5	11.69	18.5	.26	90
40-120A	(.0612)	(37.3)	20.2	22.7	39.6	.21	0
40-120B	.0612	36.3	19.65	21.8	29.6	.17	90
40-122A	(.061)	(36.1)	18.85	21.0	33.3	.21	0
40-122B	.061	36.7	18.0	21.0	27.0	.162	90
40-124A	.0585	32.3	16.0	15.05	27.5	.198	Cross Ply
40-124B	(.0585)	(31.7)	15.9	13.79	24.5	.195	

(1) Density values in parentheses are assumed to be the same as measured values of specimens from the same laminate.

(2) Chemical analyses were made for each laminate on weight basis. Values in parentheses are based on assumed density values.

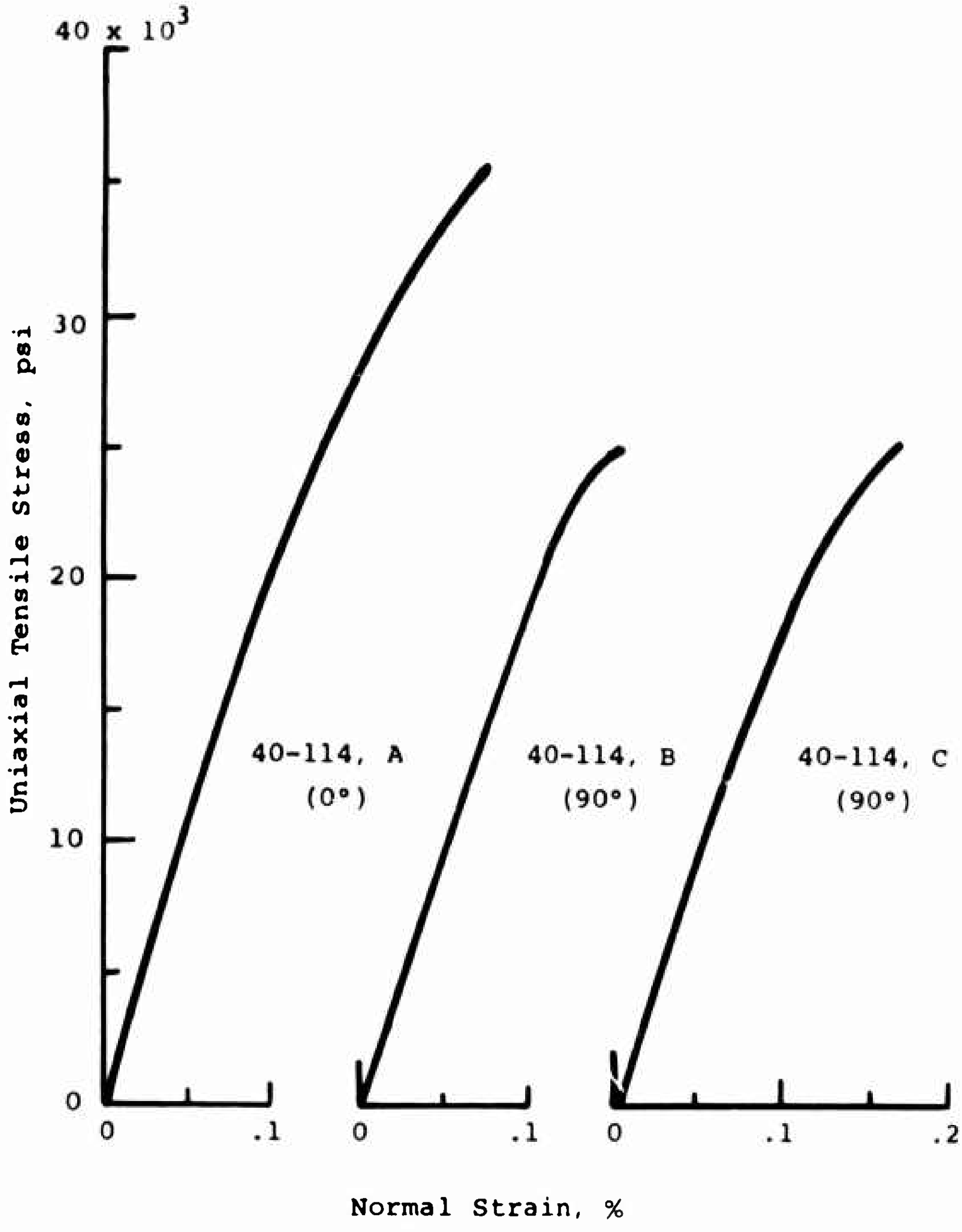

Figure 11. Tensile Stress-Strain Curves for Boron/Polyimide Laminar Composite Specimens 40-114, A (0°), B (90°), and C (90°).

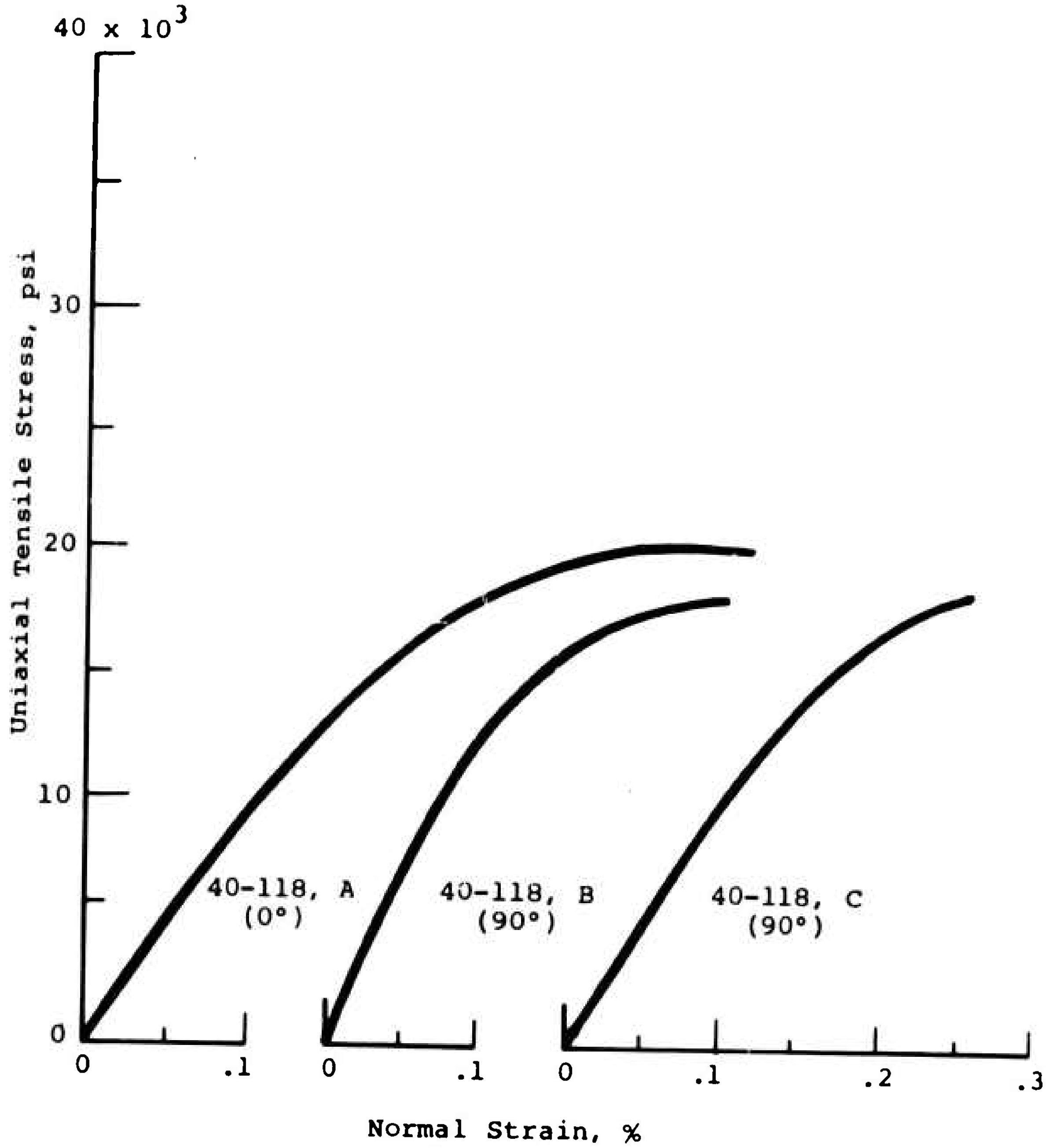

Figure 12. Tensile Stress-Strain Curves for Boron/ Polyimide Laminar Composite Specimens 40-118, A (0°), B (90°), and C (90°).

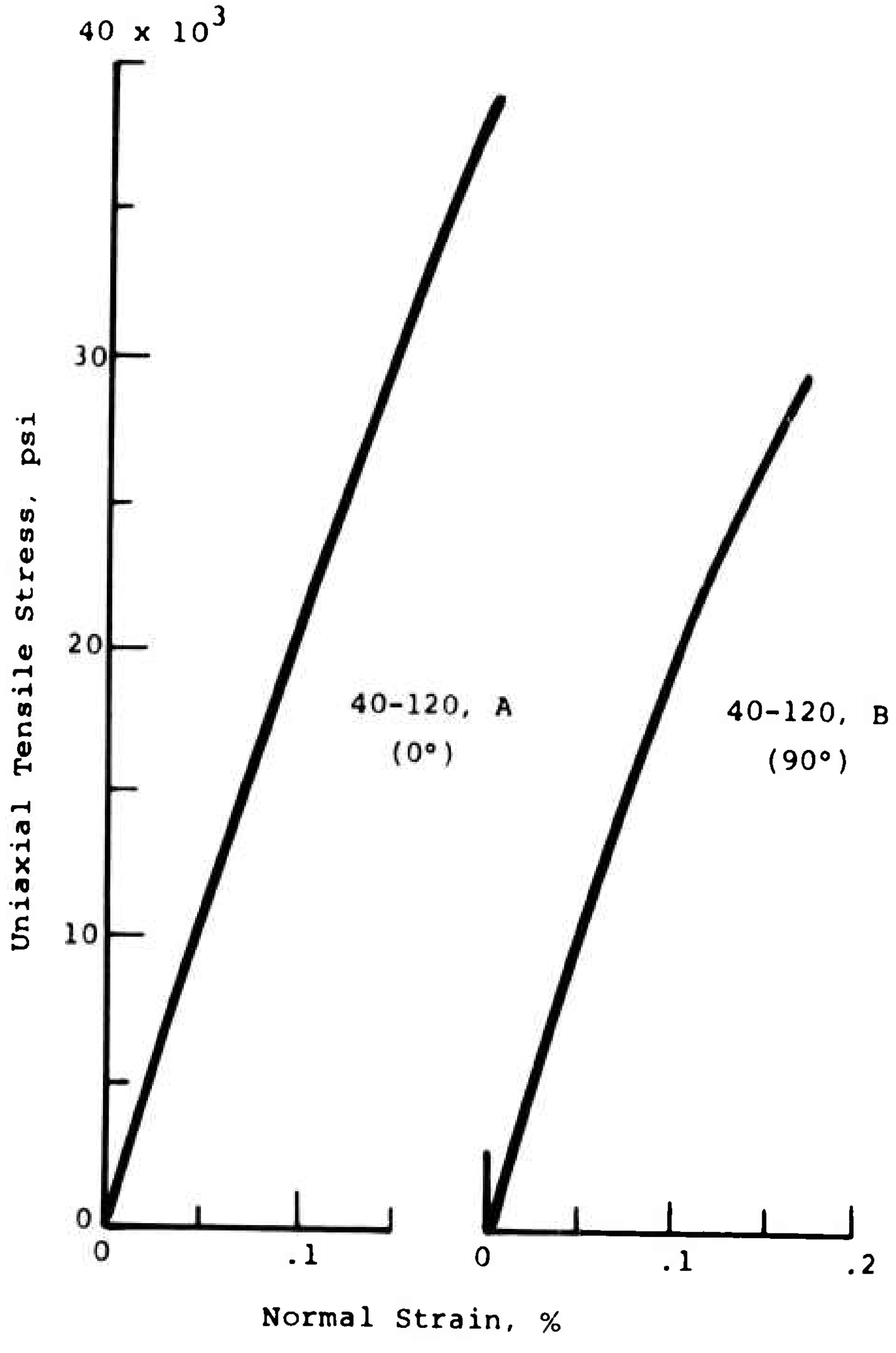

Figure 13. Tensile Stress-Strain Curves for Boron/Polyimide Laminar Composite Specimens 40-120, A (0°) and B (90°).

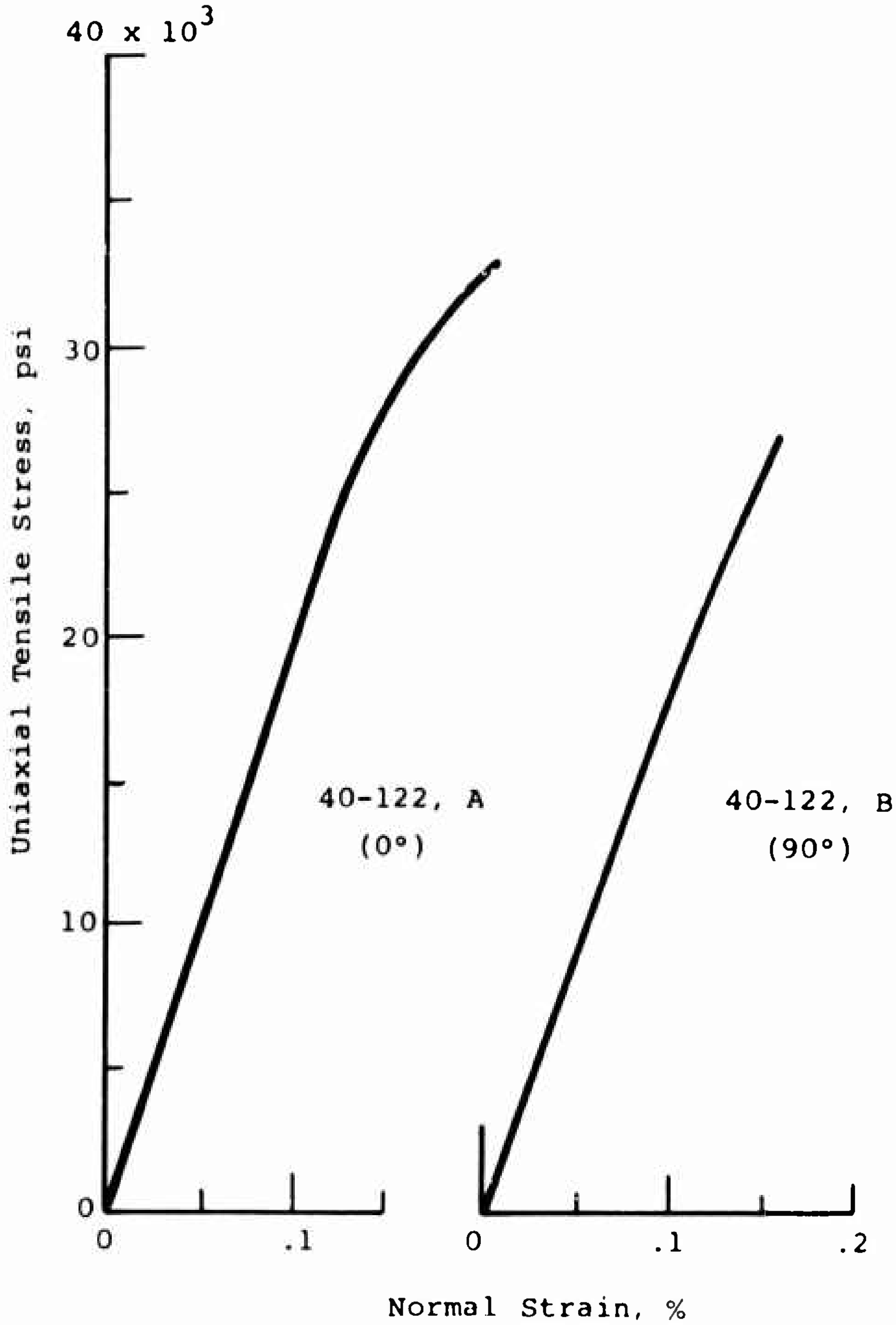

Figure 14. Tensile Stress-Strain Curves for Boron/ Polyimide Laminar Composite Specimens 40-122, A (0°) and B (90°).

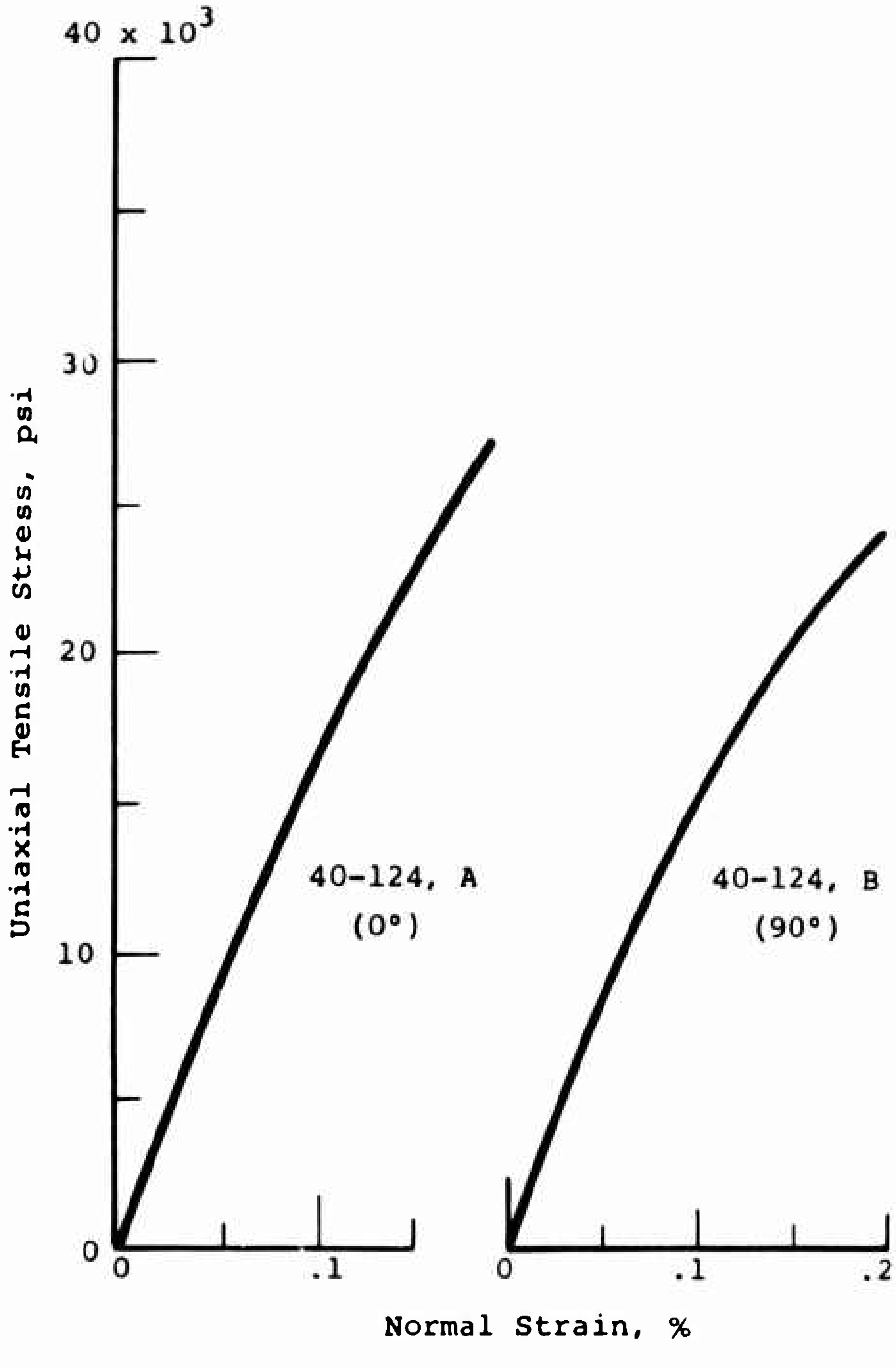

Figure 15. Tensile Stress-Strain Curves for Boron/Polyimide Laminar Composite Specimens 40-124, A (0°), and B (90°).

were similar for any one laminate. The main differences between the A, B, and C samples appeared in the plastic region of the stress-strain curve. There was, however, a noticeable exception in the case of laminate 40-118. Here, the B specimen had a modulus greater than that for A or C. Although the volume fraction of reinforcement in specimen B was higher than that in either A or C, the increment was not sufficient to explain the difference in modulus. The effective modulus of the boron in sample B was 59.7×10^6 psi as compared to 49.5×10^6 psi for A and 48.0×10^6 psi for C. The B values appear to be too high and may reflect an instrumentation error.

5. The major differences between the five laminates were related to the volume fraction of reinforcement in the composite. Since some care was taken to have the basic material (boron-coated polyimide) of uniform quality in each of these laminates, the variations in volume fraction of reinforcement were a result of variations in the thickness of adhesive used to bond the laminate layers together. Laminates with low volume fractions of boron had thick glue lines and showed the lowest mechanical properties. The higher volume fraction composites had thin glue lines and showed the highest mechanical properties.

The differences in the glue lines of laminates 40-118A and 40-120A are clearly shown in Figures 16 and 17. In these photographs, which show polished sections of the tensile specimens at the fracture zone, the white bands are boron. Adjacent to the boron is the uniform thin layer of the polyimide substrate. In some places a fine line of boron can be seen on the other side of the polyimide. This is followed by the layer of adhesive. In laminate 40-120A, the adhesive layers are very thin. In laminate 40-118A, the adhesive layers vary considerably in thickness, and in many instances they are very thick. Where the adhesive is thick, the planarity of the reinforcement is lost. Where the adhesive is thin (40-120A), the planarity appears to be good. It is likely that a high degree of planarity of the reinforcement sheets is required for high tensile properties.

In Figure 18, the tensile strengths of the laminates have been plotted against the average adhesive thickness. The figure indicates that an adhesive thickness of less than 0.2 mil is desirable. The effects of the adhesive thickness appear to be more pronounced for the A specimen than for the B and C specimens.

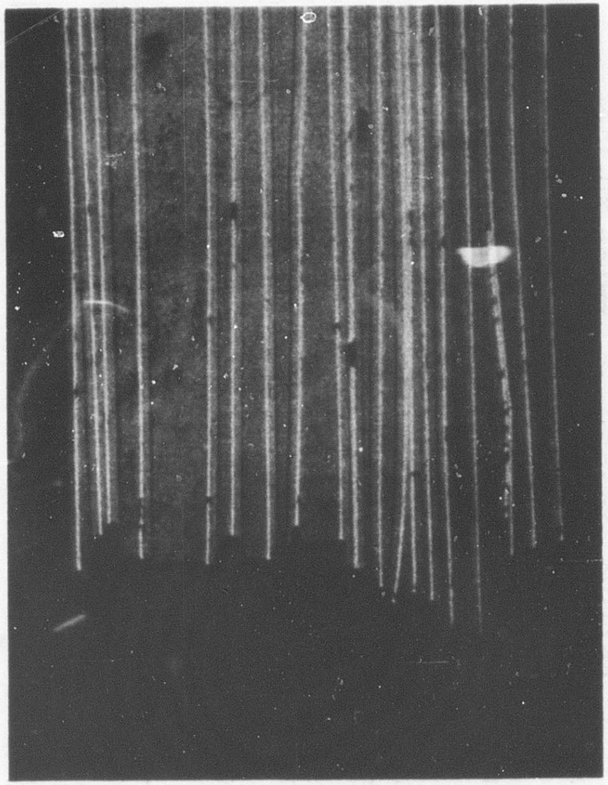

Figure 16. Polished Section at Fracture -- Specimen 40-118A, 165X.

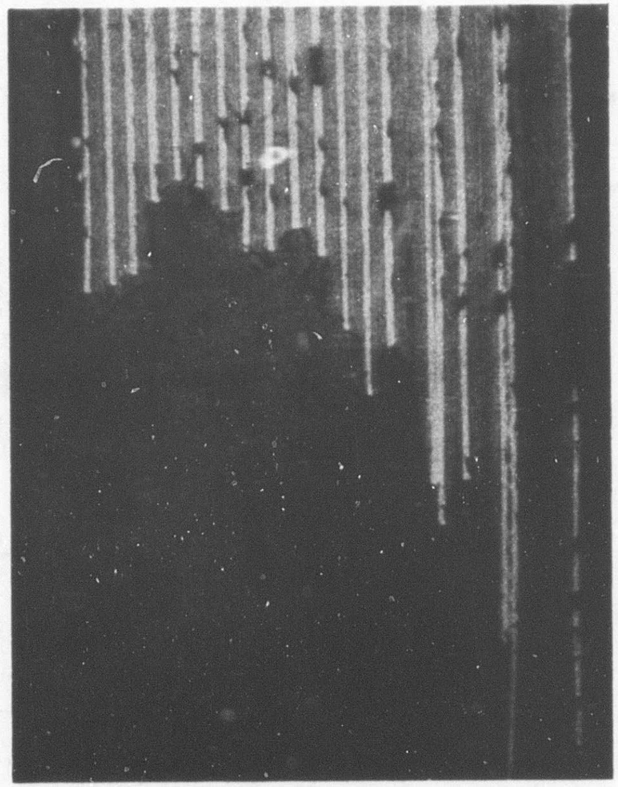

Figure 17. Polished Section at Fracture -- Specimen 40-120A, 285X.

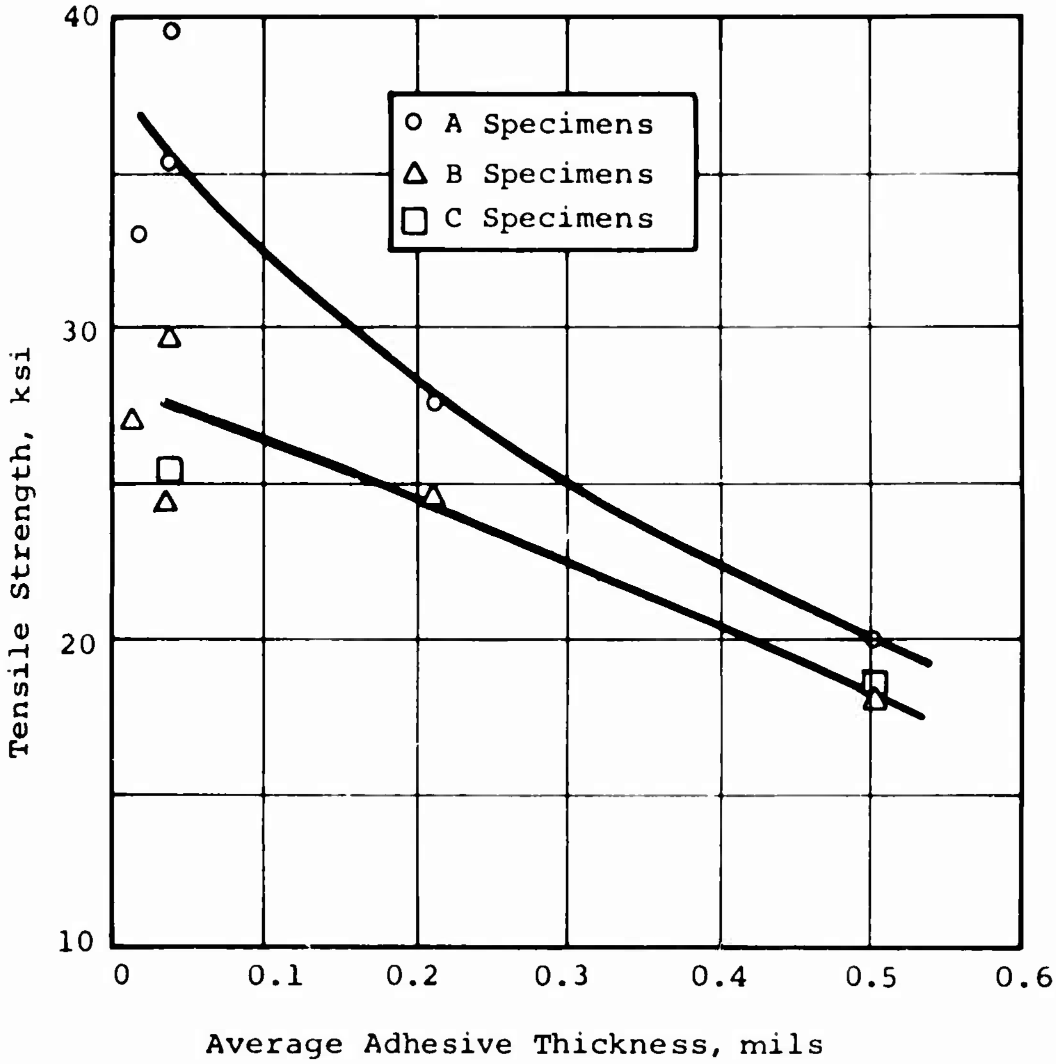

Figure 18. Effect of Adhesive Thickness on Tensile Strength.

Further work is required to regulate the adhesive thickness. The variations obtained in making the above laminates were greater than those previously experienced using highly standardized materials and techniques. One explanation for the thick adhesive layer in laminate 40-118 is that the room temperature during the lay-up of the laminate was higher than usual. This possibly indicates that more control of the whole time-temperature-pressure sequence is required to make materials with reproducible properties.

Nevertheless, the potential of these types of materials is promising. The best laminate, 40-120A, had a modulus of 20.2×10^6 psi and a tensile strength of 39.6×10^3 psi. The density was 0.061 pci. The specific modulus was therefore 3.3×10^8 inches and the specific strength was 6.5×10^5 inches. This is a material comparable to an aluminum having a strength of 65×10^3 psi and a stiffness 3.3 times that of aluminum.

CONCLUSIONS AND RECOMMENDATIONS

The use of thin plastic substrates--specifically, 1/4 mil polyimide film--resulted in significant improvements in the mechanical properties of boron reinforced composites. The optimum values obtained were modulus, 20.2×10^6 psi; tensile strength, 39.6×10^3 psi; specific modulus, 3.3×10^8 inches; and specific strength, 6.5×10^5 inches.

In general, the modulus of the composites was isotropic in the plane of the composite, as evidenced from tensile modulus measurements in the 0° and 90° directions.

Variations in the mechanical properties from laminate to laminate appeared to be primarily associated with variations in the amount of bonding adhesive incorporated in the final laminate.

Laminates with small adhesive thicknesses gave higher strength and modulus values than those with large adhesive thicknesses. Laminates which had high tensile strength showed a higher degree of planarity in the boron reinforcement.

Further work is required to determine the factors which control the thicknesses of adhesive formed in a laminate.

Methods of developing high degrees of planarity in the reinforcement need to be developed.

The work suggests that further improvements in laminate properties may result from the use of substrates even thinner than the 1/4 mil investigated in this program.

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13. ABSTRACT

Methods of making boron-plastic plane isotropic structural composites using boron film reinforcement were developed. Principal objective was to obtain high percentages of boron in the composite and values to 54% by volume were achieved. This resulted in elastic moduli up to 31×10^6 psi and tensile strengths up to 40×10^3 psi. The two fabrication techniques studied involved:

(1) a 1/4 mil polyimide substrate for the boron film,

(2) an aluminum substrate which was partially removed.

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